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Effect of Counterface Material Type and Its Topography on the Tribological Properties of Polyimide Composites

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MATERIAL TYPE AND ITS TOPOGRAPHY ON THE
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EFFECT OF COUNTERFACE MATERIAL TYPE AND ITS TOPOGRAPHY
ON THE TRIBOLOGICAL PROPERTIES OF POLYIMIDE COMPOSITES

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Abstract

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Graphite fiber reinforced polyimide composite pins were slid against seven different counterfaces to determine the effect of material type on the tribological properties of polymer composites. In addition, the effect of sliding a new pin on a pre-established transfer film was investigated. The results indicated that almost a five order of magnitude difference in composite wear rate can occur just by varying the counterface material. An attempt to make all surfaces as smooth as possible was made, but due to differences in material composition this was not possible and a range of surface roughnesses were obtained. The results indicate that the smoother the surface, the lower the composite wear rate; but that small protrusions (not discernible with arithmetic surface roughness measurements) can markedly increase wear rates. A pre-established transfer film definitely improved both run-in and steady-state wear rates.

INTRODUCTION

The need for self-lubricating materials is continuously increasing for both earth based lubrication problems and for lubrication problems that will be encountered in space. One particular class of self-lubricating materials which has demonstrated considerable promise for earth based lubrication problems is graphite-fiber-reinforced polyimide (GFRPI) (1-20). They are now being considered or already being used in various bearing, seal, gear and brake material applications (9,11-14,18-20).

The word polyimide is a generic designation and refers to a class of long-chained polymers which have repeating imide groups as an integral part of the main chain. By varying the monomeric starting materials, polyimides of different chemical composition and structure can be obtained. The polyimides as a class are characterized by a high thermal stability, and at the decomposition point they crumble to a fine powder without melting. They have a high radiation stability and can withstand exposure to neutrons, electrons, ultraviolet light, and gamma radiation. They are resistant to most common chemicals and solvents, but are attacked by alkalis.

There are many factors that can affect the tribological properties of self-lubricating composites. Some of the more common factors are temperature, speed, load, and the amount of moisture in the atmosphere. Another factor which has not been evaluated by many researchers is the effect of counterface type on the tribological properties. Some researchers (21-26) have looked at the effect of the counterface surface roughness on the tribological properties of polymers and polymer composites and mixed results have been obtained. Usually, as the counterface surface is made smoother, the wear rate of the polymer or polymer composite was found to decrease. Two exceptions were the work of Swikert and Johnson (24) and the work of Dowson, et al. (25), these studies found that there was a minimum in wear for Ultra-High-Molecular Weight Polyethylene at 0.11 and 0.03 μm respectively as the surfaces were made smoother.

The purpose of this study was to determine if improved tribological properties could be obtained with GFRPI composites by using different types of counterfaces. Six different metals were evaluated. It was attempted to make each counterface as smooth as possible, but in the process, a range of surface roughnesses and textures were obtained. To test ultimate smoothness, a pyrex

glass counterface was evaluated also. The effect of a pre-established transfer film was also investigated by conducting a test on the wear track of a previously evaluated counterface, which had a good transfer film on it.

The test conditions were a temperature of 25 °C, a sliding speed of 0.27 m/s (100 rpm), a load of 9.8 N, and a controlled atmosphere of 50 percent relative humidity. For the low wear composites the tests were run for very long sliding durations (1300 km or 55 days). Friction coefficient and wear were evaluated as a function of sliding distance for each counterface.

MATERIALS

The composite pins used in this study were made from a 50/50 (by weight) mixture of graphite fibers and polyimide resin. The fibers were chopped into lengths of 6.4×10^{-13} m (0.25 in) and were randomly dispersed throughout the polyimide matrix. The graphite fibers (designated type "L" in a previous study (15)) had a tensile strength of 6.2×10^8 N/m² and an elastic modulus of 3×10^{10} N/m².

The polyimide used as the matrix material was an addition-type polyimide and was highly crosslinked. Its molecular structure is given in Fig. 1.

Seven different disk counterface materials were evaluated. They were pyrex glass, Vitallium, Haynes 6B, 440C HT stainless steel, M-50 steel, 304 stainless steel, and René 41. Hardness and surface texture properties of these materials are given in Table I.

APPARATUS DESCRIPTION

A pin-on-disk type of tribometer was used in this study (Fig. 2). The frictions specimens consisted of a stationary (0.476 cm rad.) hemispherically tipped composite pin in sliding contact with a flat (6.3 cm diam) glass or metallic disk. The wear track diameter on the disks was 5.2 cm. The loads were applied through a lever arm, and the same lever arm transmitted the friction force to a strain gauge whose output was continuously recorded on a

strip-chart recorder. The friction specimens were enclosed in a chamber to control the atmosphere at 10 000 ppm H_2O (approximately 50 percent relative humidity at 25 °C).

SPECIMEN SURFACE FINISHING AND CLEANING

The Vitallium disks were supplied by Zimmer-USA. Their method of surface finishing was to first rough grind with 180-grit silicon carbide paper and a water coolant. The disks were then successively ground with finer grit papers of the same type until a 600-grit finish was obtained. A polishing operation followed in which 6- μm diamond paste on a nylon cloth was used with kerosene as the lubricant and coolant. The final polishing was done with a short-nap microcloth of 0.05 μm gamma alumina with distilled water as the lubricant and coolant. The final surface roughness is comparable to that used in commercial prothesis components.

The other metallic disks were lapped on a lapping wheel using commercial lapping oil and levigated alumina (submicron particle size). They were then cleaned with ethyl alcohol and polished on a polishing wheel with levigated alumina and distilled water. They were next scrubbed with a brush under running distilled water to remove the alumina and dried with clean dry compressed air.

The polyimide composite pins and pyrex glass disks were scrubbed with a nonabrasive detergent, rinsed with distilled water, and dried with clean dry compressed air.

EXPERIMENTAL TESTING

After the pin and disk specimens were inserted into the test apparatus and the chamber sealed, moist air was circulated through the chamber for 15 min before each test and continuously throughout the test. Once the chamber was purged, the disk was set into rotation at 100 rpm (0.27 m/s). The test

temperature was 25 °C and the load was 9.8 N which was gradually applied to the rotating disk through the stationary pin (Fig. 2).

At various times during the experiments the tests were stopped and the specimens removed and examined by optical microscopy. The wear scar on each pin tip was measured and the wear volume calculated. The pin was not removed from the holder, and locating pins ensured that it was returned to its original position in the apparatus.

RESULTS AND DISCUSSION

Friction Coefficient

Friction coefficient as a function of sliding distance is given in Fig. 3 for the seven different counterface materials evaluated for short sliding distances (0 to 1 km). For most of the metallic surfaces there was a tendency for the friction coefficient to drop slightly from the initial value and then to gradually increase with increasing sliding distance. Two exceptions were the M-50 stainless steel and the René 41 counterface. The friction coefficient on René 41 started out at 0.25 but it rose very quickly to 0.30 and then remained stable. The friction coefficient of M-50 tended to increase slowly with sliding distance. The pyrex glass, the smoothest surface, initially gave the highest friction coefficient (0.36), but after 1 km (1 hr) of sliding it dropped to 0.28, about the same as 440C HT stainless steel and Vitallium.

In addition to the seven different counterface materials, one test was conducted to evaluate the effect of a transfer film on the run-in friction and wear and the steady-state friction and wear. This was done by sliding a new hemispherically tipped pin on the same wear track that was used in a previous test on a Vitallium disk. The friction trace for this test is shown in Fig. 3. It is seen that by using a run-in wear track with a transfer film already deposited that the friction coefficient starts out at a slightly lower

value and tends to decrease with sliding distance rather than increase as the the nonrun-in wear track did.

Friction coefficient as a function of long sliding distances for the same counterface materials is shown in Fig. 4. After 1 km of sliding there was a variation of friction coefficient of about 0.10, but as sliding duration increased this variation tended to get greater. The highest friction coefficient was obtained with the 304 stainless steel, it tended to steadily increase with sliding duration. Most of the other metallic counterfaces tended to increase slowly as a function of sliding duration and then level off. The pyrex glass decreased slowly with sliding duration up to about 5 km of sliding and then leveled off at about 0.22. The run-in Vitallium wear track produced lower friction coefficients than the nonrun-in Vitallium wear track for the first 20 km of sliding but then equivalent values were obtained. Figure 4 only shows friction coefficients up to 100 km of sliding. The pyrex glass, and the Vitallium counterfaces were evaluated up to 1300 km of sliding and very constant friction coefficients equivalent to that found at 100 km were obtained.

Composite Wear

Wear of the composite pins was determined by stopping the tests at selected sliding intervals and measuring the wear scar on the pin tip and calculating the wear volume. Figure 5 plots these wear volume values as a function of sliding distance. In general, the wear rate (wear volume per unit sliding distance) starts out much higher than the resultant steady-state wear rate. This run-in wear rate decreases gradually with sliding distance until the steady-state value is obtained. Table II gives the sliding distance in kilometers that it took to reach the steady-state wear regime. The table also lists the steady-state wear rates obtained for each counterface which were calculated by taking a linear regression fit (least squares) of the slopes of

the lines in Fig. 5. It is interesting to note that the counterfaces which gave the lowest wear rates took the longest sliding time to reach that steady-state value.

To obtain a measure of run-in wear, wear rates were calculated for 0 to 100 m of sliding. Those values are also given in Table II. The lowest run-in wear rates were obtained with the Vitallium disk on the run-in wear track. The smoothest surface, pyrex glass, gave the next lowest run-in rates, with the Vitallium, Haynes 6B, 440C HT and 304 stainless steel giving equivalent values. M-50 stainless steel was about twice as high as the previous group and René 41 about 10 times higher. Comparing these data to the surface texture parameters of Table I, it is seen that the M-50 stainless steel and the René 41 had a higher average roughness (R_a) value than the others and the René 41 had a much higher maximum peak-to-valley height than the others. More will be discussed about this in a later section.

The lowest steady-state wear rates were obtained with the pyrex glass disk counterface and the run-in Vitallium disk counterface. The value being an extremely low value of $0.007 \times 10^{-15} \text{ m}^3/\text{m}$ of sliding. The next best was the Vitallium disk counterface with the value being $0.02 \times 10^{-15} \text{ m}^3/\text{m}$ of sliding. This was an order of magnitude lower than the next best disk counterface, Haynes 6B; and two orders of magnitude better than 440C HT, 304, and M-50 steels. René 41 gave a wear rate of $24 \times 10^{-15} \text{ m}^3/\text{m}$ of sliding which was much higher than any of the other counterface disk surfaces. The 304 stainless steel disk counterface produced a change on the wear rate after 44 km of sliding. As will be discussed in a following section, this has been attributed to galling of the counterface.

Composite Wear Surface Morphology

During the intervals the tests were stopped to measure the wear scar diameters, optical microscopy studies of the wear surfaces were conducted.

Most of the composite wear surfaces were worn very smooth and there was a sharp demarcation between the graphite fibers and the polyimide matrix (Fig. 6(a)). For short sliding distances all the composites which slid on metallic counterfaces, except the René 41 counterface, looked like the counterface shown in Fig. 6(a). The René 41 counterface produced a wear surface that was very rough and was a mixture of graphite and polyimide back-transferred material (Fig. 6(b)). It was impossible to discern fibers on the surface.

The Vitallium (both new and run-in) and Haynes 68 counterfaces produced composite wear surfaces similar to that shown in Fig. 6(a) for long sliding durations (up to 1300 km). The M-50 counterface did also but after about 10 km of sliding the composite surfaces became covered with bands of back-transferred material (Fig. 7(b)). There was a corresponding band found on the counterface wear track (Fig. 7(a)). This effect also happened for 440C HT stainless steel but it occurred after about 84 km of sliding.

The pyrex glass counterfaces produced slightly different types of composite wear surfaces. For very short sliding distances the surfaces looked like that of Fig. 6(a), but as sliding continued a very thin back transferred material was observed on the surfaces. Figure 8(a) shows this thin back-transferred material after 46 km of sliding. On further sliding this back-transferred film tended to get thicker and eventually completely covered the fibers. Figure 8(b) shows the surface after 600 km of sliding. This film is different than the other types of back-transfer observed in that it was uniformly distributed across the scar and appeared to be much thinner. Also there was no matching band of heavy transfer on the counterface.

Counterface Transfer Films

For short sliding distances the transfer film on all the metallic counterfaces consisted of plastically flowing thin platelets (Fig. 9(a)); as

sliding continued the platelets coalesced together to form a thin, uniform layer of transferred material. Interference bands could be observed in the material indicating it was from 0.4 to 0.8 μm thick (Fig. 9(b)). By far the most continuous thin layers were obtained on the Vitallium counterface with the Haynes 6B being the next best. The steel counterfaces also produced good transfer films, but on long sliding durations the transfer tended to build-up on certain areas of the wear track to produce thick bands of heavy transfer (Fig. 7(a)). Generally when this happened higher friction and higher wear occurred.

For the first 44 km of sliding the 304 stainless steel counterface gave good wear results (even though friction tended to increase steadily with sliding distance). But at about 44 km of sliding the composite pin caused galling of the 304 counterface to occur. Figure 10 gives a surface profile of the wear track showing the galling which took place. The galling tended to cause the composite wear-rate to increase about three times and the corresponding area on the composite (to that of the galled area on the counterface wear track) looked like the surface obtained with the René 41 counterface. That is it was very rough, grooved and looked "plowed like."

The René 41 counterface produced the highest composite wear rates. The wear track on this counterface was found to be quite different from the others. Apparently what happened was when the surfaces were lapped and polished there was a tendency to remove the soft phase of the material and not to remove the hard carbide particles. Thus a surface was produced which had very small protrusion sticking up on the surface which then abraded the composite counterface. Figure 11 gives a photomicrograph of the René 41 counterface wear track showing the transfer film and the carbide protrusions. Figure 12(g) gives a surface profile of the counterface showing that these

protrusions extended about 1 μm above the surface. The transfer tended to build-up around the protrusions (Fig. 11) but this did not mitigate the wear.

Photomicrographs of the wear tracks on the pyrex disk is shown in Fig. 13 after 1 and 850 km of sliding. As opposed to the metallic counterfaces there does not appear to be any transfer film, at least not like those observed with the metallic surfaces. No attempt was made to analytically determine if there were a transfer film present; however optical observations at high magnifications indicated that there was a milky colored film present. The film was thinner than 0.4 μm (wavelength minimum of visible light) since it was too thin for interference bands to be observed.

Surface Texture Effects

Figure 11 gives surface profiles of the seven different disk counterfaces used in the study, and Table I gives some selected surface texture parameters of the disks. Five of the surfaces show a negative skewness, only 440C HT stainless steel and M-50 steel were positive. Comparing the data of Table I and II, it appears positively skewed surfaces tend to give higher wear, although an exception is the René 41 surface which showed negative skewness but very high wear. Also 304 stainless steel gave high negative skewness and relatively high wear. In the case of René 41, the skewness did not take into account that there were sharp protrusions on the René 41 surface, the valleys more than balanced them out. This points out the fact that more than one parameter must be taken into account to adequately characterize a surface.

Even though it is felt that more than one parameter is necessary to characterize a surface, Fig. 14 plots composite pin wear rate as a function of arithmetic mean surface roughness, and Fig. 15 plots composite pin wear rate as a function of the maximum height of surface profile above the mean line. Such a wide variation of the data was obtained that the data had to be plotted on semilog paper. For both arithmetic average and maximum height of the

profile above the mean line, the wear rate tends to decrease steadily with decreasing surface roughness and decreasing maximum height of the surface profile until at some point there is a very rapid drop. For the arithmetic mean surface roughness, this occurred in the range of 0.03 to 0.05 μm and for the maximum height above the mean line it occurred about 0.2 to 0.3 μm . The one exception was 304 stainless steel, the wear obtained for it was higher than the next higher value on each curve. It is believed that the reason for the higher wear with this material was that it was in the process of galling, so an additional parameter in addition to original surface texture was involved.

Ignoring protrusions and galling, wear rate tends to decrease as surfaces are made smoother. This is in contrast to what some others have found in that they believe there is an optimum value of surface roughness which promotes transfer and minimum wear (24,25). Most of the data obtained on the effect of surface roughness has been done using the same material and applying different surface finishes to it. The author has found that by trying to polish a metal with a hard phase in it, to obtain a very smooth surface finish, often times you remove the soft phase of the material and leave behind the hard phase. An R_a value of the surface may indicate it is smoother than the less polished surface, but it can contain the protrusions which increase wear. René 41 was the prime example of this; and although the data is not presented, a less polished René 41 surface provided lower wear because the protrusions were absent. It was also found that a similar situation could occur with 440C HT stainless steel, but it had to be polished for a very long period of time for this to happen because of its hardness.

Friction coefficient does not seem to correlate with surface roughness. It tends to be more dependent on sliding duration and the establishment of a transfer film. Depending on the type of transfer film the friction will either stabilize or increase with sliding duration.

CONCLUDING REMARKS

The results of this study indicate that the smoother the counterface material the lower the steady-state wear rate of the composite pin material. One exception was the 304 stainless steel counterface, but it galled after a certain sliding duration, thus another parameter may have entered into the results. Glass gave the lowest wear rate and was the smoothest surface. However glass is a hydrated surface, and this may have been beneficial to the wear of the graphite fibers.

The results also indicate that if a transfer film can somehow be pre-established on a surface, lower run-in and steady-state wear can be obtained. In addition, lower run-in friction coefficients are also obtained but the friction coefficient eventually reaches a value equivalent to the surface without the pre-established transfer film.

The smoothness of the surfaces, as measured by arithmetic mean surface roughness (R_a), definitely appeared to have an effect on the wear rate of the GFRPI composite, but the study indicates that this parameter may not necessarily indicate the presence of small protuberance that act as a very abrasive surface. Thus values such as R_p , maximum height of the profile above the mean line, or R_t , maximum peak-to-valley height of the profile, are important in characterizing a counterface. A surface profile trace is also helpful in accessing the state of the surface.

SUMMARY OF RESULTS

Tribological studies on six different metallic surfaces, a pyrex glass surface, and a previously run-in Vitallium wear track indicate that:

1. Up to almost five order of magnitude differences in the GFRPI composite wear rate were obtained by sliding it against different counterface materials.

2. Pyrex glass surfaces gave the lowest values of friction coefficient (0.21) and steady-state wear rates ($0.007 \times 10^{-15} \text{ m}^3/\text{m}$ of sliding). The Vitallium surface with the pre-established transfer film on the wear track gave equivalent steady-state wear rates but higher friction coefficients (0.29).

3. The smoother the counterface surface the lower the composite steady-state wear rate. No correlation between the surface finish and friction coefficient was observed.

4. Metallic protrusions on the surface markedly increased wear, therefore at least two parameters should be used to characterize surfaces, R_a and R_p .

5. The lowest friction coefficients and wear values were obtained when very thin transfer films or back-transfer films were obtained.

6. The build-up of thick bands of transfer, usually accompanied by thick back-transfer bands, tended to increase friction coefficients and wear rates.

7. Smoother surfaces tended to give a lower run-in wear rate, but it took a longer time to reach a steady-state wear rate.

8. The 304 stainless steel surface galled after a period of sliding and increased the wear rate of the composite. Friction coefficient on this counterface steadily increased as a function of sliding distance.

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TABLE I. - HARDNESS AND SURFACE TEXTURE PARAMETERS OF THE DISK SURFACES

Surface parameters	Pyrex glass	Vitallium	Haynes 6-B	440C-HT stainless steel	M-50 steel	304 stainless steel	René 41
Rockwell hardness	-----	C-34	C-44	C-58	C-62	B-84	C-35
R_a (μm)	0.006	0.016	0.050	0.056	0.11	0.035	.12
R_{sk}	-1.4	-.88	-.60	+.80	+.40	-1.3	-.10
R_t (μm)	0.078	0.39	0.65	0.75	1.6	0.41	2.6
R_p (μm)	0.039	0.11	0.22	0.35	1.0	0.20	1.5
S_m (μm)	1800	1300	150	430	150	580	390
H_{sc}	1	3	25	13	25	6	10

R_a - Arithmetic mean of the departures of the profile from the mean line.

R_{sk} - Skewness, a measure of the asymmetry of the amplitude distribution curve about the mean line.

R_t - The maximum peak-to-valley height of the profile.

R_p - The maximum height of the profile above the mean line.

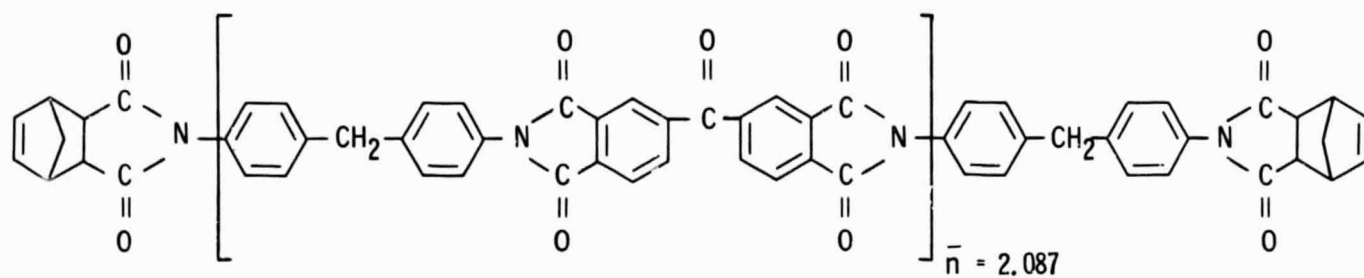
S_m - The mean spacing between profile peaks at the mean line.

H_{sc} - The number of complete profile peaks projecting above the mean line.

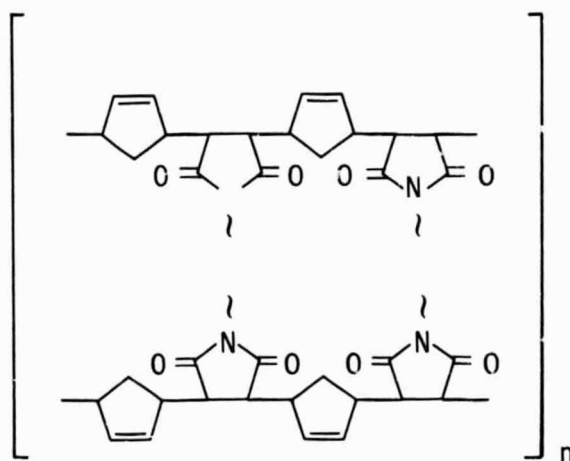
TABLE II. - SUMMARY OF WEAR RATE RESULTS

Counterface material	Total sliding time, hr	Run-in (-0.1 km) wear rate (m^3/m of sliding)	Sliding distance to reach steady-state wear, km	Steady-state wear rate (m^3/m of sliding)
Pyrex glass	1310	1.7×10^{-15}	46	$0.007 \pm 0.002 \times 10^{-15}$
Vitallium	1024	3.6×10^{-15}	50	$0.02 \pm 0.01 \times 10^{-15}$
Vitallium (run-in wear track)	773	0.91×10^{-15}	62	$0.007 \pm 0.003 \times 10^{-15}$
Haynes 6B	203	2.9×10^{-15}	2	$0.24 \pm 0.10 \times 10^{-15}$
440C HT	114	3.5×10^{-15}	0.1	$2.8 \pm 1.5 \times 10^{-15}$
M-50 steel	110	8.6×10^{-15}	0.1	$4.4 \pm 1.0 \times 10^{-15}$
304 stainless steel	65	4.2×10^{-15}	1	$2.3 \pm 1.0 \times 10^{-15}$
René 41	10	51×10^{-15}	0.1	$24 \pm 4 \times 10^{-15}$

^aAfter 44 hr of sliding wear rate increased to 7.0×10^{-15} due to galling of the 304 stainless steel.



(a) Imidized prepolymer.



(b) Cured polyimide (idealized structure).

Figure 1. - Addition-type of polyimide polymer (type "A") used in this study.

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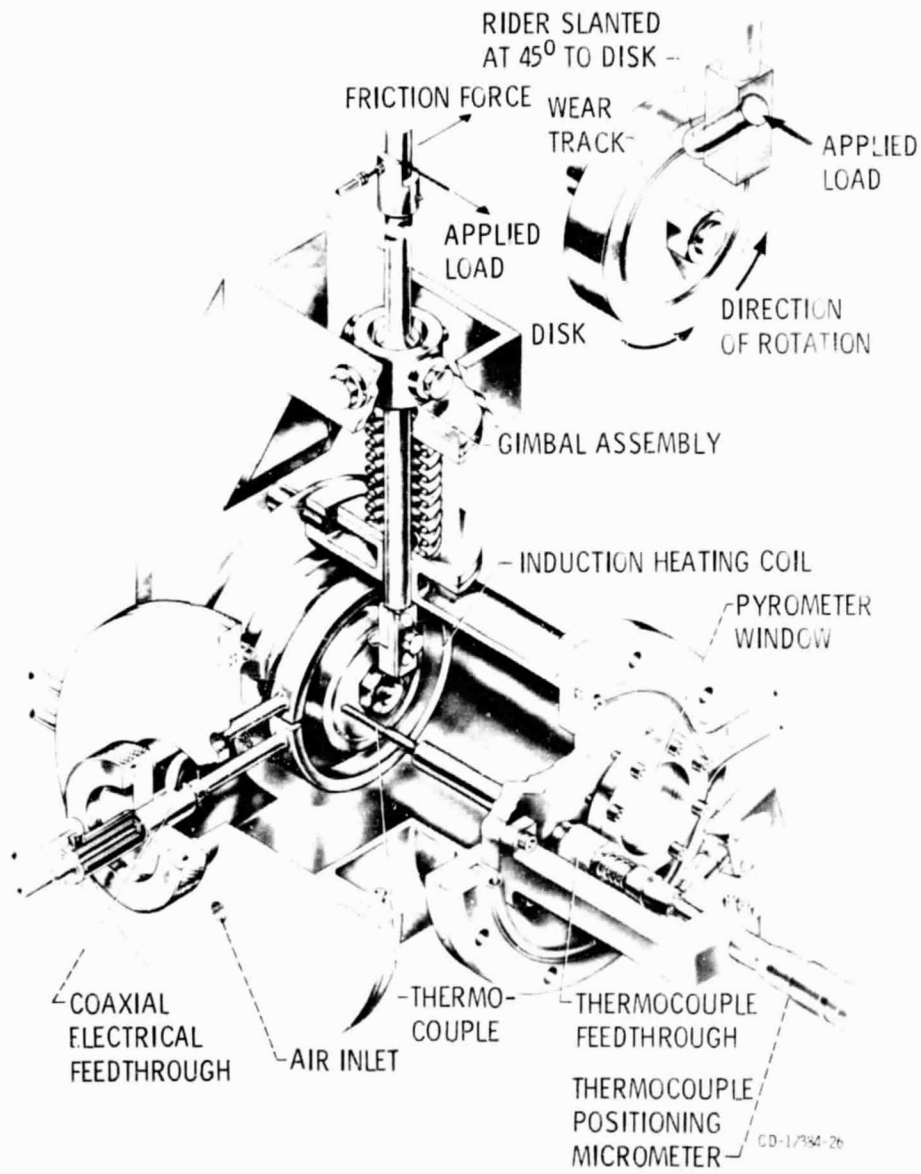


Figure 2. - Pin-on-disk tribometer.

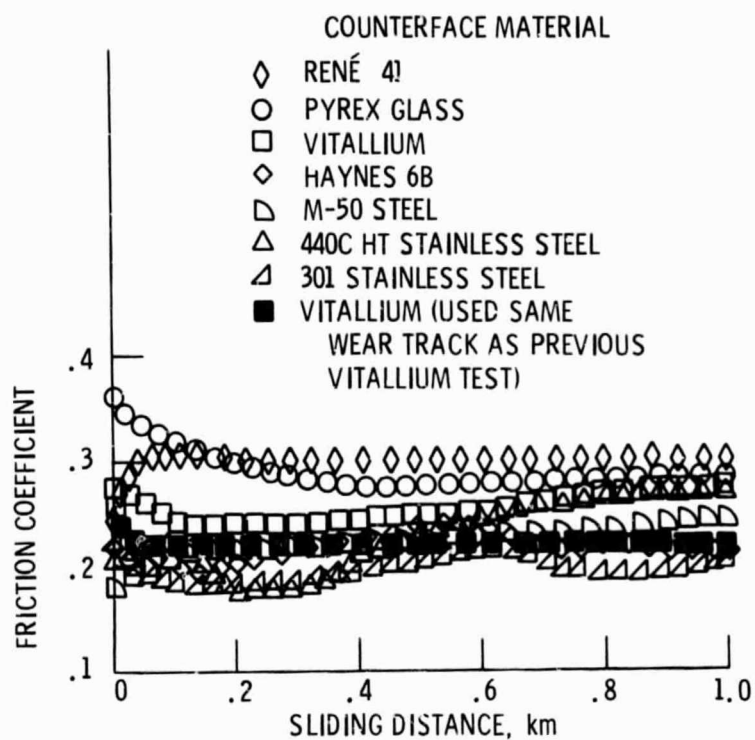


Figure 3. - Friction coefficient as a function of sliding distance for short sliding distances.

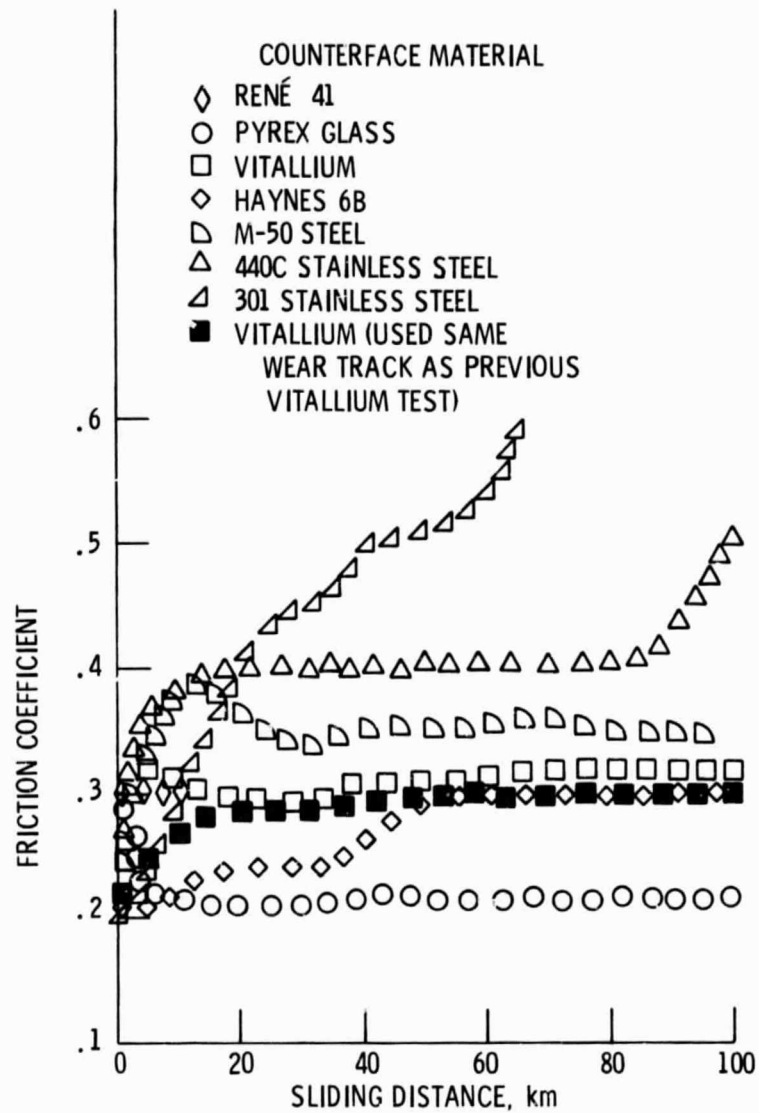


Figure 4. - Friction coefficient as a function of sliding distance for long sliding distances.

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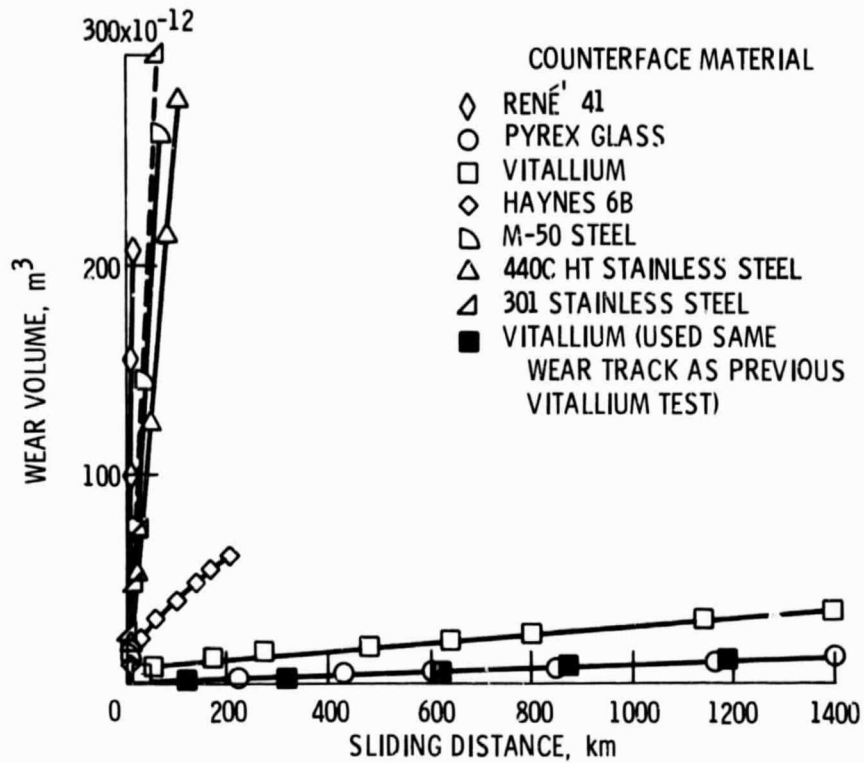


Figure 5. - Wear volume of graphite fiber reinforced polyimide pins sliding against different counterfaces as a function of sliding distance.

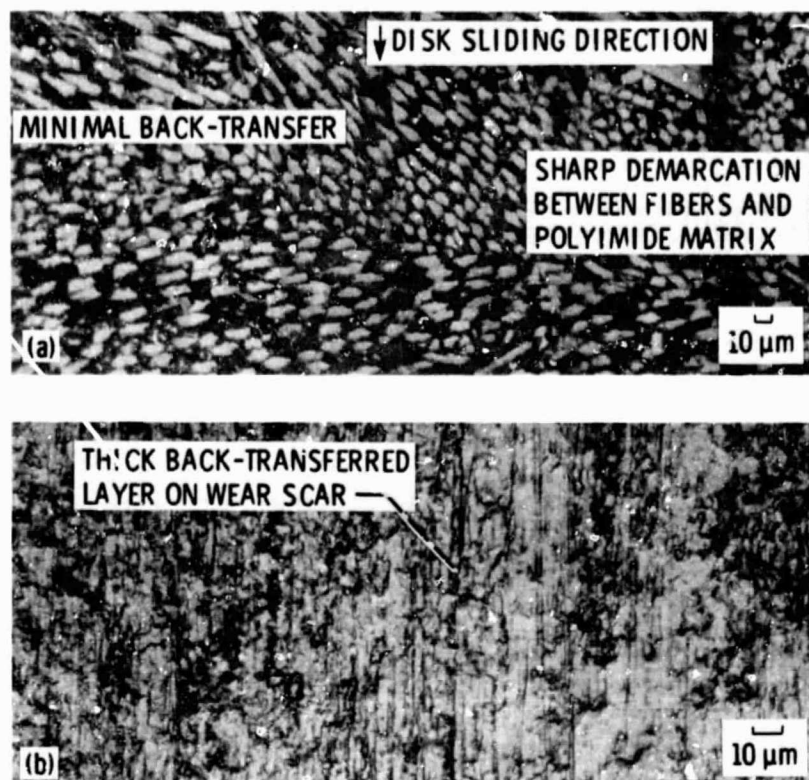


Figure 6. - Photomicrographs of graphite fiber reinforced polyimide pin wear surfaces which slid against (a) Vitallium for 300 kilometers of sliding, and (b) René 41 for 1 kilometer of sliding.

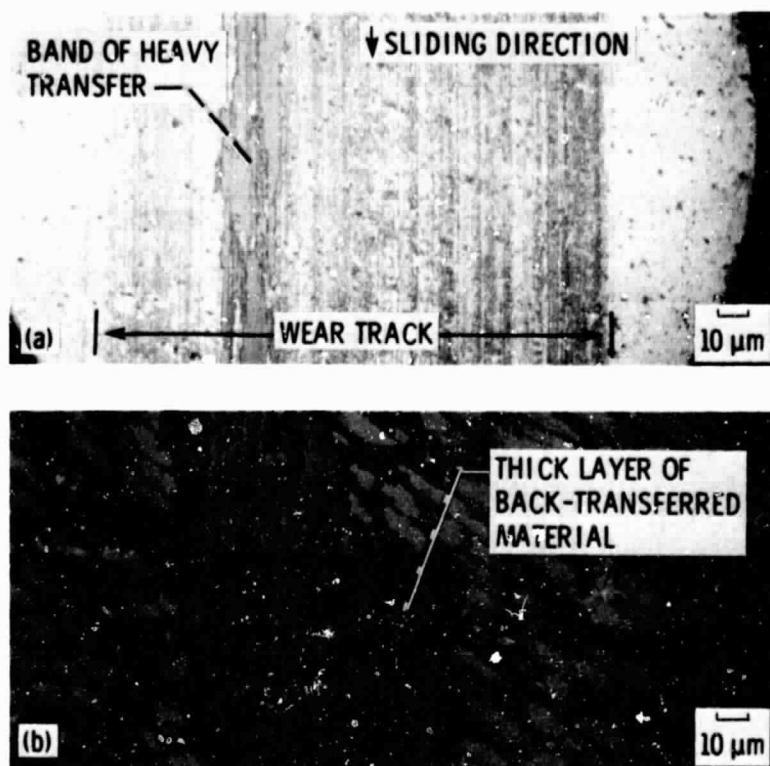


Figure 7. - Photomicrographs of (a) the wear track on the M-50 steel disk, and (b) the wear scar on the GFRPI pin after 40 kilometers of sliding.

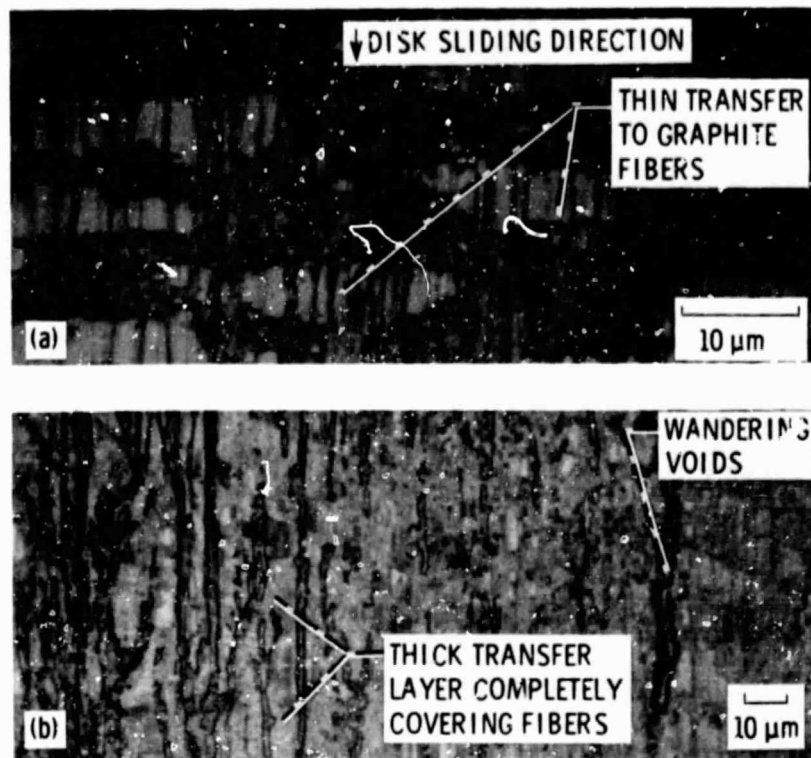


Figure 8. - Photomicrographs of graphite fiber reinforced polyimide pin wear surfaces which slid against pyrex disks after (a) 46, and (b) 600 kilometers of sliding.

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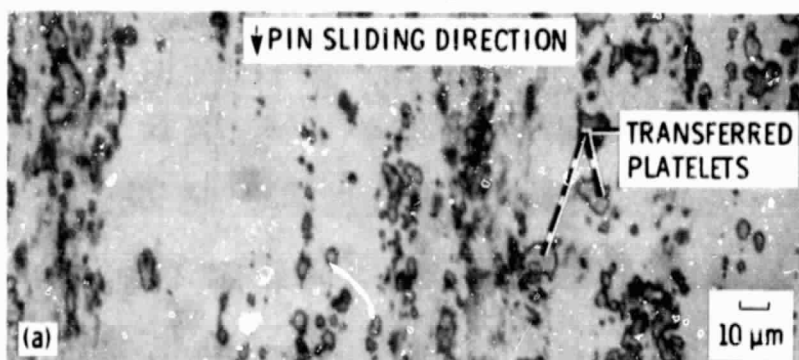


Figure 9. - Photomicrographs of transfer to the vitallium disk after (a) 1 kilometer, and (b) 500 kilometer of sliding.



Figure 10. - Surface profile of the wear track on 304 stainless steel disk showing the galling type of wear that took place.

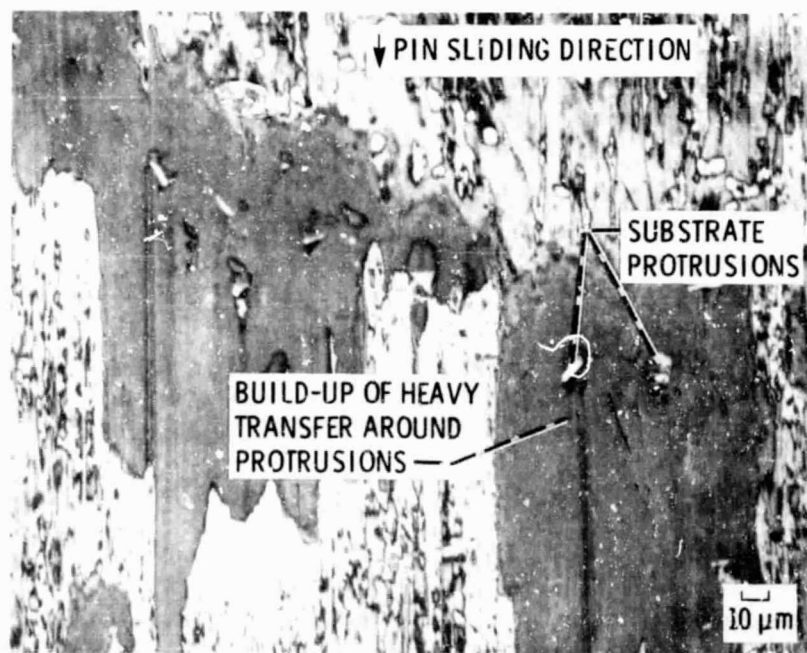


Figure 11. - Photomicrographs of transfer to the René 41 disk after 1 kilometer of sliding.

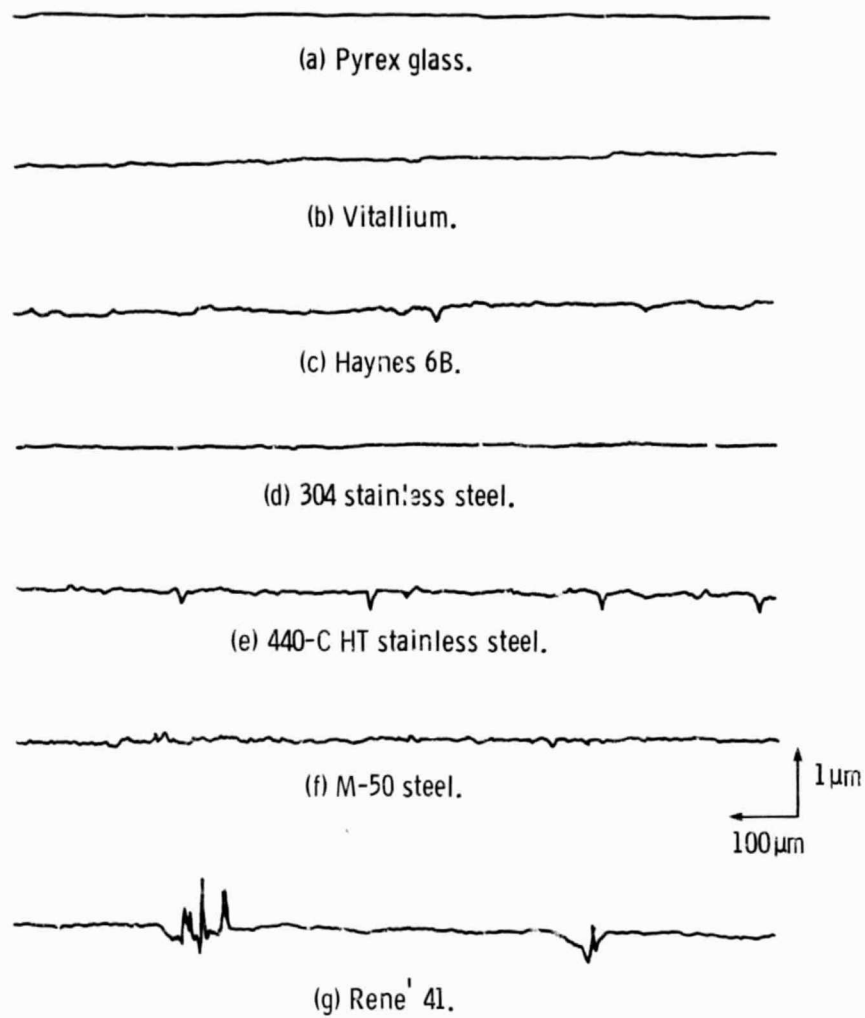


Figure 12. - Surface profiles of disks used in this study.

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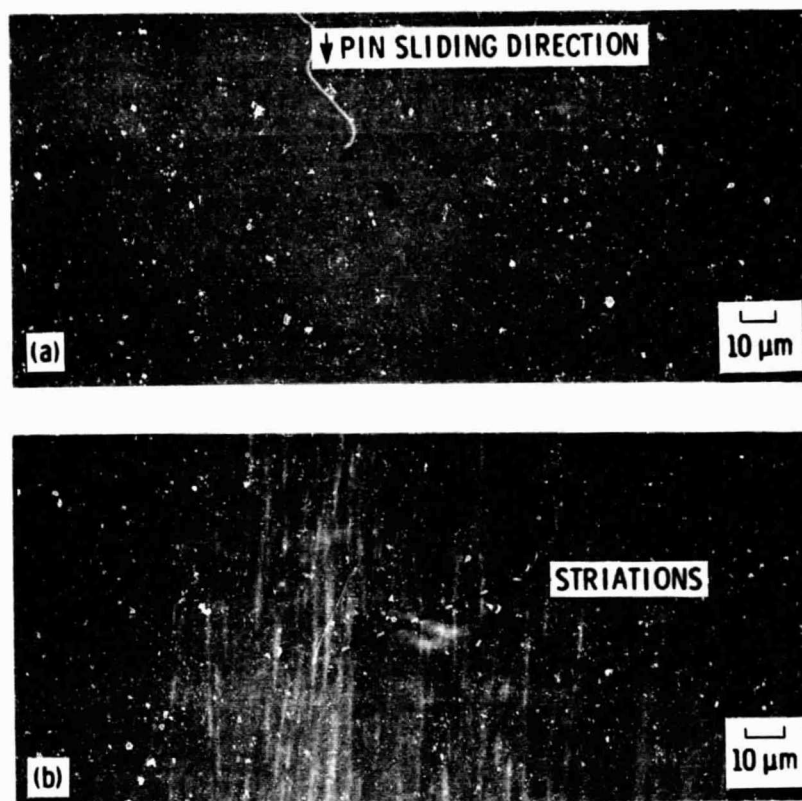


Figure 13. - Photomicrographs of transfer to pyrex disk
after (a) 1 kilometer, and (b) 850 kilometers of sliding.

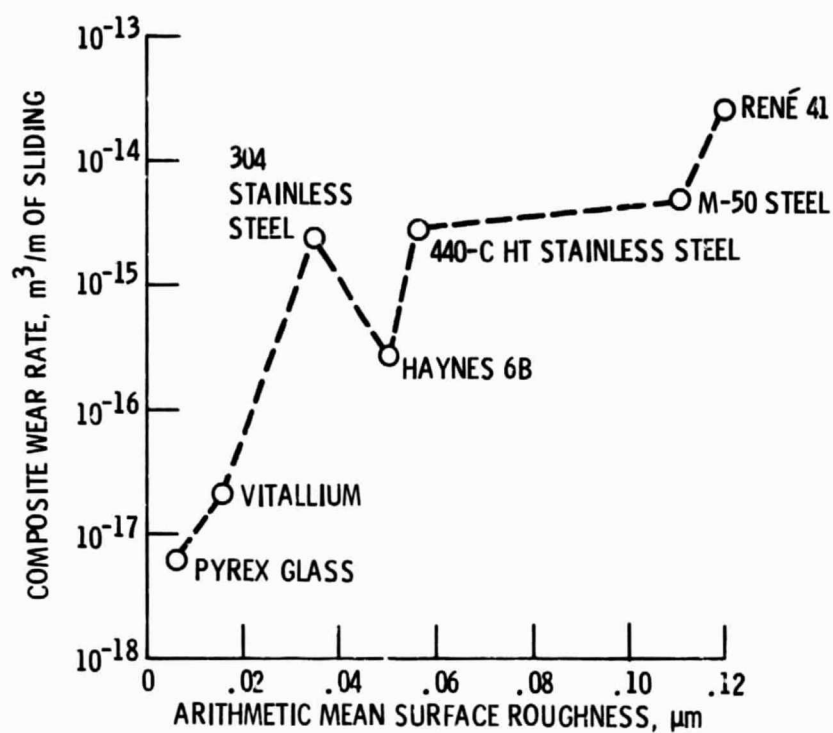


Figure 14. - Composite pin wear rate as a function of arithmetic mean surface roughness.

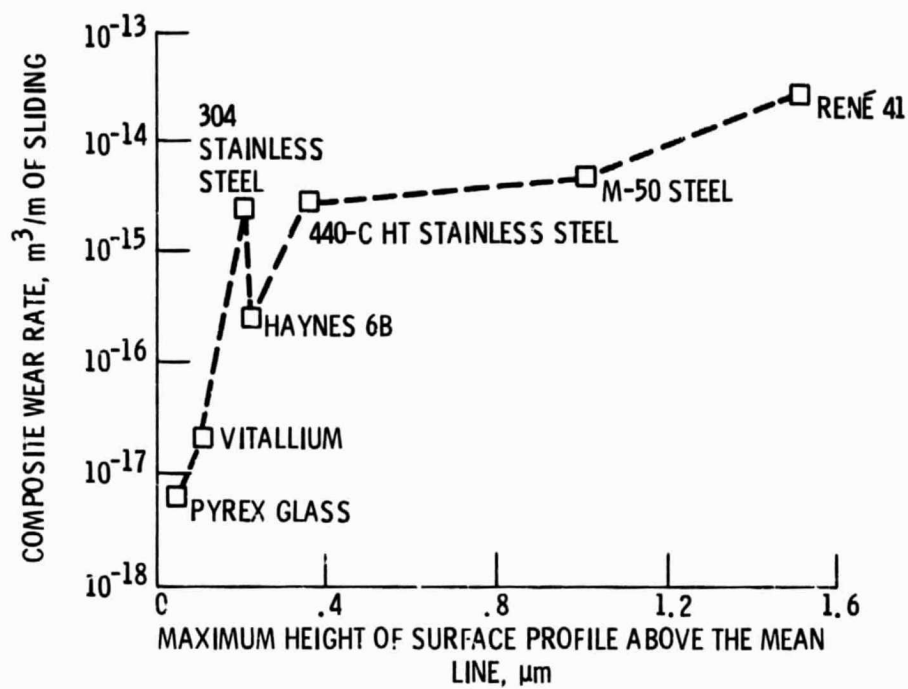


Figure 15. - Composite pin wear rate as a function of the maximum height of the surface profile above the mean line.

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16. Abstract Graphite fiber reinforced polyimide composite pins were slid against seven different counterfaces to determine the effect of material type on the tribological properties of polymer composites. In addition, the effect of sliding a new pin on a pre-established transfer film was investigated. The results indicated that almost a five order of magnitude difference in composite wear rate can occur just by varying the counterface material. An attempt to make all surfaces as smooth as possible was made, but due to differences in material composition this was not possible and a range of surface roughnesses were obtained. The results indicate that the smoother the surface, the lower the composite wear rate; but that small protrusions (not discernible with arithmetic surface roughness measurements) can markedly increase wear rates. A pre-established transfer film definitely improved both run-in and steady-state wear rates.					
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